

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE		ATTORNEY'S DOCKET NUMBER
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371		4197-116
INTERNATIONAL APPLICATION NO.		U.S. APPLICATION NO. (If known, see 37 CFR 1.5) <b>10/089143</b>
PCT/DE00/03409		INTERNATIONAL FILING DATE 29 September 2000
		PRIORITY DATE CLAIMED 06 October 1999, 15 October 1999
TITLE OF INVENTION <b>METHOD AND DEVICE FOR REGULATING THE COMPOSITION OF SOLUTION(S)</b>		
APPLICANT(S) FOR DO/EO/US <b>NIEMZ, Frank-Gunter; MEYER, Wilhelm; MAINZER-ALTHOF, Tanja</b>		
Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:		
<ol style="list-style-type: none"> <li>1. <input checked="" type="checkbox"/> This is a <b>FIRST</b> submission of items concerning a filing under 35 U.S.C. 371.</li> <li>2. <input type="checkbox"/> This is a <b>SECOND</b> or <b>SUBSEQUENT</b> submission of items concerning a filing under 35 U.S.C. 371.</li> <li>3. <input type="checkbox"/> This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).</li> <li>4. <input checked="" type="checkbox"/> A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.</li> <li>5. <input checked="" type="checkbox"/> A copy of the International Application as filed (35 U.S.C. 371(c)(2))             <ol style="list-style-type: none"> <li>a. <input checked="" type="checkbox"/> is transmitted herewith (required only if not transmitted by the International Bureau).</li> <li>b. <input checked="" type="checkbox"/> has been transmitted by the International Bureau.</li> <li>c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US).</li> </ol> </li> <li>6. <input checked="" type="checkbox"/> A translation of the International Application into English (35 U.S.C. 371(c)(2)).</li> <li>7. <input checked="" type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))             <ol style="list-style-type: none"> <li>a. <input type="checkbox"/> are transmitted herewith (required only if not transmitted by the International Bureau).</li> <li>b. <input type="checkbox"/> have been transmitted by the International Bureau.</li> <li>c. <input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired.</li> <li>d. <input checked="" type="checkbox"/> have not been made and will not be made.</li> </ol> </li> <li>8. <input type="checkbox"/> A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).</li> <li>9. <input checked="" type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).*(<b>Unsigned</b>)</li> <li>10. <input type="checkbox"/> A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).</li> </ol>		
<b>Items 11. to 16. below concern other document(s) or information included:</b>		
<ol style="list-style-type: none"> <li>11. <input type="checkbox"/> An Information Disclosure Statement under 37 CFR 1.97 and 1.98.</li> <li>12. <input type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.</li> <li>13. <input checked="" type="checkbox"/> A FIRST preliminary amendment. <input type="checkbox"/> A SECOND or SUBSEQUENT preliminary amendment.</li> <li>14. <input type="checkbox"/> A substitute specification.</li> <li>15. <input checked="" type="checkbox"/> A small entity statement.</li> <li>16. <input type="checkbox"/> Other items or information:</li> </ol>		

NOTE: This application is being filed with an unsigned Oath or Declaration under the provisions of 37 CFR § 1.53 in order that applicants may secure a filing date of March 26, 2002. Upon receipt of a "Notice to File Missing Parts - Filing Date Granted," an executed Declaration and Power of Attorney, will be filed in the Patent and Trademark Office. The undersigned agent affirmatively states that she has been duly authorized and appointed to file this application on behalf of the applicants and that the Declaration and Power of Attorney to be filed hereafter will confirm the undersigned agent's authorization and appointment. Applicants are entitled to small entity status within the meaning of 37 CFR § 1.9.

10/089143

17. <input checked="" type="checkbox"/> The following fees are submitted: <b>Basic National Fee</b> (37 CFR 1.492(a)(1)-(5)): Search Report has been prepared by the EPO or JPO ..... \$860.00  International preliminary examination fee paid to USPTO (37 CFR 1.482) ..... \$0.00 No International preliminary examination fee paid to USPTO (37 CFR 1.482) but international search fee paid to USPTO (37 CFR 1.445(a)(2)) ..... \$0.00  Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO ..... \$1000.00  International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(2)-(4) ..... \$0.00				CALCULATIONS	PTO USE ONLY																								
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Surcharge of <b>\$130.00</b> for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e)).				<b>\$</b>																									
<table border="1" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th style="text-align: left;">Claims</th> <th style="text-align: left;">Number Filed</th> <th style="text-align: left;">Number Extra</th> <th style="text-align: left;">Rate</th> <th colspan="2"></th> </tr> </thead> <tbody> <tr> <td>Total Claims</td> <td>16 -20 =</td> <td>0</td> <td>X \$18.00</td> <td style="text-align: center;"><b>\$</b></td> <td></td> </tr> <tr> <td>Independent Claims</td> <td>3- 3 =</td> <td>0</td> <td>X \$80.00</td> <td style="text-align: center;"><b>\$</b></td> <td></td> </tr> <tr> <td colspan="3">Multiple dependent claim(s) (if applicable)</td> <td>+ \$270.00</td> <td style="text-align: center;"><b>\$</b></td> <td></td> </tr> </tbody> </table>						Claims	Number Filed	Number Extra	Rate			Total Claims	16 -20 =	0	X \$18.00	<b>\$</b>		Independent Claims	3- 3 =	0	X \$80.00	<b>\$</b>		Multiple dependent claim(s) (if applicable)			+ \$270.00	<b>\$</b>	
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Reduction by 1/2 for filing by small entity, if applicable. Verified Small Entity statement must also be filed. (Note 37 CFR 1.9, 1.27, 1.28).				<b>\$ 430.00</b>																									
<b>SUBTOTAL =</b>				<b>\$ 430.00</b>																									
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<b>TOTAL NATIONAL FEE =</b>				<b>\$ 430.00</b>																									
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). <b>\$40.00</b> per property +				<b>\$</b>																									
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				<b>refunded</b>																									
				<b>Charged</b>	<b>\$</b>																								
a. <input checked="" type="checkbox"/> A check in the amount of <u>\$430.00</u> to cover the above fees is enclosed.  b. <input type="checkbox"/> Please charge my Deposit Account No. _____ in the amount of \$ _____ to cover the above fees. A duplicate copy of this sheet is enclosed.  c. <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. <u>08-3284</u> . A duplicate copy of this sheet is enclosed.																													
<b>NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not yet been met, a petition to revive (37 CFR 1.127(a) or (b)) must be filed and granted to restore the application to pending status.</b>																													
SEND ALL CORRESPONDENCE TO: <b>Steven J. Hultquist</b> <b>Intellectual Property/Technology Law</b> <b>P. O. Box 14329</b> <b>Research Triangle Park, NC 27709</b>																													
 <b>MARIANNE FUIERER</b> <b>Registration No. 39,983</b>																													

10/089143

JC13 Rec'd PCT/PTO 26 MAR 2002

4197-116  
PATENT APPLICATION

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

**In re Application of:** Niemz, et al.

**Application No.:** New U.S. National Stage Application of  
PCT International Application No. PCT/DE00/03409

**International Filing Date:** 29 September 2000

**Priority Date Claimed:** 06 October 1999 (German Appl. No. 199 47 908.9)  
15 October 1999 (German Appl. No. 199 49 727.3)

**U.S. National Phase Filing Date:** Date of mailing identified below

**Title:** **METHOD AND DEVICE FOR REGULATING THE  
COMPOSITION OF SOLUTION(S)**

**EXPRESS MAIL CERTIFICATE**

I hereby certify that I am mailing the attached documents to the  
Commissioner for Patents on the date specified, in an envelope  
addressed to the Commissioner for Patents, Box Patent  
Application, Washington, DC 20231, and Express Mailed under  
the provisions of 37 CFR 1.10.

  
23448  
PATENT & TRADEMARK OFFICE

\_\_\_\_\_  
Blake Crouch

\_\_\_\_\_  
Name of Person Mailing This Document

  
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Signature

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March 26, 2002

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Express Mail Label Number

**PRELIMINARY AMENDMENT**

Commissioner for Patents  
BOX PATENT APPLICATION  
Washington, D.C. 20231

Sir:

Prior to examination of the above-identified new national phase patent application, please amend the application, as follows:

**In the Specification**<sup>1</sup>

Please insert on page 1, between the title of the application and the first paragraph, the following new paragraph:

**CROSS-REFERENCE TO RELATED APPLICATIONS**

This application is filed under the provisions of 35 U. S.C. §371 and claims the priority of International Patent Application No. PCT/DE00/03409 filed September 29, 2000, which in turn claims priority of German Patent Application Nos. 199 47 908.9 filed October 6, 1999 and 199 49 727.3 filed October 15, 1999.

**Field of the Invention**

On page 2, between the first and second paragraph, please insert the following:

**BACKGROUND OF THE INVENTION**

On page 3, between the first and second paragraph, please insert the following:

**DESCRIPTION OF THE INVENTION**

**In the Claims**

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<sup>1</sup> Applicants has provided a marked-up version of amended paragraphs, and claims 1-7 in Appendix A, and a clean set of all pending claims, amended to date, in Appendix B.

1. A process for the regulation of the composition of solution(s) for the manufacture of cellulosic mouldings, comprising:

(a) mixing cellulose pulp and a water containing aminoxide to form a cellulosic suspension;

(b) evaporating water from the cellulosic suspension to form a cellulose solution;

(c) extruding the cellulose solution through an air gap into an precipitation bath, which contains an aqueous aminoxide solution wherein the cellulose solution coagulates to form mouldings;

(d) conducting the form mouldings through an aqueous washing solution in which remaining aminoxide is washed out from the mouldings; and

(e) removing at least some of the aqueous aminoxide solutions from stage (c) and/or the (d) for measuring a non-optical property and for regulating the composition of the measured solutions according to the deviation of the measured value of at least one non-optical property from a given reference value for a reference-composition wherein the measured non-optical property of the solution is selected from the group consisting of: dielectricity constant, inductive conductivity, microwave absorption, density, water content and ultrasonic speed.

2. The process according to claim 1, wherein the temperature of the solution is measured at or shortly before or after the measurement of the property and the measured value is compensated on the basis of the measured temperature.

3. The process according to claim 1, wherein the property is measured in-line.

4. The process according to claim 1, wherein adjustments are made to the components of the solutions in stage (a), (c), or (d).

5. The process according to claim 1, wherein adjustments are made to the operating conditions in stage (b) and/or (e) for the regulation of the composition of the solutions.

6. A device used for the regulation of the composition of solution(s) for the manufacture of cellulosic mouldings, comprising:

a mixing apparatus containing at least two metering elements for introduction of composition components;

a dissolving and evaporation device communicatively connected to the mixing apparatus;

an extrusion device communicatively connected to the dissolving and evaporation device;

a precipitation bath downstream of the extrusion device and separated therefrom by an air gap;

at least one washing bath downstream from the precipitation bath;

a line connected between the washing bath and at least one of the metering devices, wherein the line further comprises a return evaporator;

a plurality of devices for the measurement of a non-optical property and arranged to communicate with metering elements, the evaporator device and/or return evaporator via at least one regulation circuit for the regulation of the composition of the solutions contained in the mixing apparatus, the dissolving and evaporation device and/or the precipitation bath.

7. The device according to claim 6, wherein the measuring device further comprises a device for measuring the temperature of the solution and for compensating the measured values of the device according to the temperature.

Please add the following new claims 8-16.

8. The process according to claim 2, wherein the non-optical property is measured in-line.

9. The process according to claim 1, wherein the solution from step (d) is concentrated before the non-optical property is measured.

10. The process according to claim 3, further comprising measuring the temperature of the solutions of steps (c) and/or (d) at a time selected from the group consisting of; before the measuring of the non-optical property, after the measuring of the non-optical property, and at approximately the same time as measuring the non-optical property, and wherein the measured value is compensated on the basis of the measured temperature.

11. The device of claim 6, further comprising a return line from the washing bath connected to the precipitation bath.

12. The device of claim 11, further comprising a measuring device positioned between the washing bath and precipitation bath.

13. The device of claim 7, wherein the measuring device measures a measurement selected from the group consisting of:

a dielectricity constant, inductive conductivity, microwave absorption, density, water content and ultrasonic speed.

14. The device of claim 13, wherein each measuring device is measuring a different property.

15. The process according to claim 1, wherein the aqueous aminoxide solutions from stage (c) and (d) are measured for a non-optical property.

16. A process for the regulation of the composition of solution(s) for the manufacture of cellulosic mouldings, comprising:

- (a) mixing cellulose pulp and a water containing aminoxide to form a cellulosic suspension[,];
- (b) evaporating water from the cellulosic suspension to form a cellulose solution;
- (c) extruding the cellulose solution through an air gap into an precipitation bath, which contains an aqueous solution wherein the cellulose solution coagulates to form mouldings;
- (d) conducting the form mouldings through an aqueous washing solution in which remaining aminoxide is washed out from the mouldings; and
- (e) removing at least some of the aqueous solutions from stage (c) and (d) for measuring a non-optical property and for regulating the composition of the measured solutions according to the deviation of the measured value of at least one non-optical property from a given reference value for a reference-composition wherein the measured non-optical property of the solution is selected from the group consisting of: dielectricity constant, inductive conductivity, microwave absorption, density, water content and ultrasonic speed.

## REMARKS

It is requested that the examination and prosecution of this application proceed on the basis of these amended and new claims 1-16

Respectfully submitted,



Marianne Fuierer

Registration No. 39,983

Attorney for Applicants

INTELLECTUAL PROPERTY/  
TECHNOLOGY LAW  
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Fax: (919) 419-9354  
Attorney File: 4197-116

## APPENDIX A

### In the Specification

Please insert on page 1, between the title of the application and the first paragraph, the following new paragraph:

#### --CROSS-REFERENCE TO RELATED APPLICATIONS

This application is filed under the provisions of 35 U. S.C. §371 and claims the priority of International Patent Application No. PCT/DE00/03409 filed September 29, 2000, which in turn claims priority of German Patent Application Nos. 199 47 908.9 filed October 6, 1999 and 199 49 727.3 filed October 15, 1999.

Field of the Invention--

On page 2, between the first and second paragraph, please insert the following:

#### --BACKGROUND OF THE INVENTION--

On page 3, between the first and second paragraph, please insert the following:

#### --DESCRIPTION OF THE INVENTION--

### In the Claims

Please amend claims 1 - 7 to read as follows:

1. A process [Process] for the regulation of the composition of solution(s) for the manufacture of cellulosic mouldings, [in which] comprising:

(a) mixing cellulose pulp and a water containing aminoxide [are mixed under formation of] to form a cellulosic suspension[,];

(b) evaporating water from the cellulosic suspension to form a cellulose solution [is formed from the suspension under the evaporation of water,];

(c) extruding the cellulose solution [is extruded] through an air gap into an precipitation bath, which contains an aqueous aminoxide solution wherein the cellulose solution [, and there] coagulates to form mouldings[,];

(d) conducting the form mouldings [are conducted] through an aqueous washing solution in which remaining aminoxide is washed out from the mouldings; and

(e) removing at least some of the [leads] aqueous aminoxide solutions from stage (c) and/or the (d) for measuring a non-optical property and [after concentration back to stage (a), in this a property of the solutions is measured] for regulating [and according to the measured value] the composition of the measured solutions [is regulated, characterized in that,] according to the deviation of the measured value of at least one non-optical property [or physical behavior of at least one of the said solutions is measured and the deviation(s) from the measured value respectively values] from a given reference value for a reference-composition [is used for the regulation of the composition(s) of these solutions, in which] wherein the measured non-optical property of the solution is selected from the group consisting of: [chosen from the] dielectricity constant, [the] inductive conductivity, microwave absorption, [the] density, [the] water content and [the] ultrasonic speed.

2. The process [Process] according to claim 1, wherein [characterized in that,] the temperature of the solution is

measured at or shortly before or after the measurement of the property and the measured value is compensated on the basis of the measured temperature.

3. The process [Process] according to claim 1, wherein [claims 1 to 2, characterized in that,] the property is measured in-line.

4. The process [Process] according to claim 1, wherein [claims 1 to 3, characterized in that,] adjustments are made to the components [recourse is made to the regulation of the composition] of the solutions in [the metering of the components in] stage (a), (c), or (d).

5. The process [Process] according to claim 1, wherein [claims 1 to 4, characterized in that, recourse is] adjustments are made to the operating conditions in stage (b) and/or (e) for the regulation of the composition of the solutions.

6. A device [Device] used for the regulation of the composition of solution(s) for the manufacture of cellulosic mouldings, comprising:

[for the performance of the process according to any of the claims 1 to 5, with]

a mixing apparatus [(2)] containing at least two metering elements for introduction of composition components; [(1a, 1b),]

[the mixing apparatus connected to] a dissolving and evaporation device [(3),] communicatively connected to the mixing apparatus;

an extrusion device [(4)] communicatively connected [via line 8] to the dissolving and evaporation device [(3),]; [with]

a [downstream] precipitation bath [(5),] downstream of the extrusion device and separated therefrom by an air gap; at least one washing bath downstream form the precipitation bath; [, which contains a diluted aqueous aminoxide solution, and]

a line connected between the washing bath and at least one of the metering devices, wherein the line further comprises a return evaporator;

a plurality of devices for the measurement of a non-optical property and arranged to communicate [and of the solution(s) to control their composition,

characterized in that, that in the line (8, 19, 24) or in a container, which contains the solution to be controlled, a device (9, 21, 11) for the measurement of a non-optical property of the solution is arranged, and that the measuring device together ] with metering elements [(1a, 1b, 17) or the evaporation devices (3, 20)] evaporator device and/or return evaporator via at least one [forms] regulation circuit[s (13, 14, 15, 18, 23)] for the regulation of the composition of the solutions contained in the mixing apparatus, the dissolving and evaporation device and/or the precipitation bath.

7. The device [Device] according to claim 6, wherein [characterized in that,] the measuring device [(9, 11, 21)] further comprises a device for measuring the temperature of the solution and for compensating the measured values of the device according to the temperature.

**APPENDIX B****In the Specification**

Please insert on page 1, between the title of the application and the first paragraph, the following new paragraph:

**CROSS-REFERENCE TO RELATED APPLICATIONS**

This application is filed under the provisions of 35 U. S.C. §371 and claims the priority of International Patent Application No. PCT/DE00/03409 filed September 29, 2000, which in turn claims priority of German Patent Application Nos. 199 47 908.9 filed October 6, 1999 and 199 49 727.3 filed October 15, 1999.

**Field of the Invention**

On page 2, between the first and second paragraph, please insert the following:

**BACKGROUND OF THE INVENTION**

On page 3, between the first and second paragraph, please insert the following:

**DESCRIPTION OF THE INVENTION****Pending Claims 1-16**

1. A process for the regulation of the composition of solution(s) for the manufacture of cellulosic mouldings, comprising:

- (a) mixing cellulose pulp and a water containing aminoxide to form a cellulosic suspension;
- (b) evaporating water from the cellulosic suspension to form a cellulose solution;

(c) extruding the cellulose solution through an air gap into an precipitation bath, which contains an aqueous aminoxide solution wherein the cellulose solution coagulates to form mouldings;

(d) conducting the form mouldings through an aqueous washing solution in which remaining aminoxide is washed out from the mouldings; and

(e) removing at least some of the aqueous aminoxide solutions from stage (c) and/or the (d) for measuring a non-optical property and for regulating the composition of the measured solutions according to the deviation of the measured value of at least one non-optical property from a given reference value for a reference-composition wherein the measured non-optical property of the solution is selected from the group consisting of: dielectricity constant, inductive conductivity, microwave absorption, density, water content and ultrasonic speed.

2. The process according to claim 1, wherein the temperature of the solution is measured at or shortly before or after the measurement of the property and the measured value is compensated on the basis of the measured temperature.

3. The process according to claim 1, wherein the property is measured in-line.

4. The process according to claim 1, wherein adjustments are made to the components of the solutions in stage (a), (c), or (d).

5. The process according to claim 1, wherein adjustments are made to the operating conditions in stage (b) and/or (e) for the regulation of the composition of the solutions.

6. A device used for the regulation of the composition of solution(s) for the manufacture of cellulosic mouldings, comprising:

a mixing apparatus containing at least two metering elements for introduction of composition components;

a dissolving and evaporation device communicatively connected to the mixing apparatus;

an extrusion device communicatively connected to the dissolving and evaporation device;

a precipitation bath downstream of the extrusion device and separated therefrom by an air gap;

at least one washing bath downstream from the precipitation bath;

a line connected between the washing bath and at least one of the metering devices, wherein the line further comprises a return evaporator;

a plurality of devices for the measurement of a non-optical property and arranged to communicate with metering elements, the evaporator device and/or return evaporator via at least one regulation circuit for the regulation of the composition of the solutions contained in the mixing apparatus, the dissolving and evaporation device and/or the precipitation bath.

7. The device according to claim 6, wherein the measuring device further comprises a device for measuring the temperature of the solution and for compensating the measured values of the device according to the temperature.

8. The process according to claim 2, wherein the non-optical property is measured in-line.

9. The process according to claim 1, wherein the solution from step (d) is concentrated before the non-optical property is measured.

10. The process according to claim 3, further comprising measuring the temperature of the solutions of steps (c) and/or (d) at a time selected from the group consisting of; before the measuring of the non-optical property, after the measuring of the non-optical property, and at approximately the same time as measuring the non-optical property, and wherein the measured value is compensated on the basis of the measured temperature.

11. The device of claim 6, further comprising a return line from the washing bath connected to the precipitation bath.

12. The device of claim 11, further comprising a measuring device positioned between the washing bath and precipitation bath.

13. The device of claim 7, wherein the measuring device measures a measurement selected from the group consisting of: a dielectricity constant, inductive conductivity, microwave absorption, density, water content and ultrasonic speed.

14. The device of claim 13, wherein each measuring device is measuring a different property.

15. The process according to claim 1, wherein the aqueous aminoxide solutions from stage (c) and (d) are measured for a non-optical property.

16. A process for the regulation of the composition of solution(s) for the manufacture of cellulosic mouldings, comprising:

- (a) mixing cellulose pulp and a water containing aminoxide to form a cellulosic suspension[,];
- (b) evaporating water from the cellulosic suspension to form a cellulose solution;
- (c) extruding the cellulose solution through an air gap into an precipitation bath, which contains an aqueous solution wherein the cellulose solution coagulates to form mouldings;
- (d) conducting the form mouldings through an aqueous washing solution in which remaining aminoxide is washed out from the mouldings; and
- (e) removing at least some of the aqueous solutions from stage (c) and (d) for measuring a non-optical property and for regulating the composition of the measured solutions according to the deviation of the measured value of at least one non-optical property from a given reference value for a reference-composition wherein the measured non-optical property of the solution is selected from the group consisting of: dielectricity constant, inductive conductivity, microwave absorption, density, water content and ultrasonic speed.

1/PYD

PROCESS AND DEVICE FOR CONTROLLING THE COMPOSITION OF  
SOLUTION(S)

The invention relates to a process for controlling the composition of solution(s) in the production of cellulosic shaped bodies, in which (a) cellulose pulp and an aminoxide containing water are mixed under formation of a suspension, (b) a cellulose solution is formed from the suspension under the evaporation of water, (c) the cellulose solution is extruded through an air gap into an aqueous precipitation bath and there coagulates to form mouldings, (d) the form mouldings were conducted through an aqueous washing solution, in which remaining aminoxide is washed out of the form mouldings, and (e) aqueous aminoxide solutions from stage (c) and/or (d) after concentration leads back to stage (a), in which a property or physical behavior of the solution is measured, and on the basis of the measured value the composition of the solutions are regulated.

The invention also relates to a device for the performance of this process with a mixing apparatus equipped with metering elements, a dissolving and evaporation device connected to the mixing apparatus, and an extrusion device connected via a line to the dissolving and evaporation device, with a downstream precipitation bath, at least one washing bath, containing a strongly diluted, aqueous aminoxide solution, a device for the measurement of one property of the solutions with the purpose of controlling their composition. When mention is made in general in the present invention of a property to be measured of the cellulose solution, this should also be understood to include a measurable physical behaviour of the solution.

The properties of the cellulosic mouldings manufactured in accordance with the aminoxide process, in particular the textile physical properties of the spun fibres and filaments, depend to a large degree on the composition of the extrusion solution. In order to maintain the optimum properties it is therefore desirable for the composition of the solution to be monitored and for fluctuations to be kept within narrow limits. In a continuous process like the present Aminoxide-Process its monitoring is of importance for an economic running. It is important, to measure also the concentration of the aminoxide solutions in stage (c), (d) and (e) and to regulate the composition of the solution.

From WO 94/28212 the principle is known of taking a sample from time to time of the spinning solution flowing to the extrusion apparatus, and of measuring its refractive index, which at 60 °C should lie in the range from 1.4890 to 1.491, in order to obtain extrusion products with satisfactory properties. It is also known to keep the refractive index of the aminoxide solution in certain limits. Thereafter the refractive index of the aqueous aminoxide solution of the precipitation bath at 60 °C could lie in a range of 1,3644 to 1,3708, whereas for the concentrated aminoxide solution for the formation of the cellulose suspension the refractive index could lie in a range from 1,4620 to 1,4628, without activation of the metering elements or changes in the evaporation conditions. The monitoring of the refractive index of the solution in a more or less wide range does not allow the exact regulation of the composition of the solution. In addition this process does not work in case of solutions with strong lightabsorbtion and /or -diffraction.

2a

The EP 0254 803 shows a process for the production of an aqueous, pure N-Methylmorpholine-N-oxide-solution by reaction of Methylmorpholine with an aqueous Hydrogen peroxide, in which mixtures of Methylmorpholin and water were destillated, the acetotrop of Methylmorpholine and Water at temperatures from 60°C to 100°C was reacted with an aqueous Hydrogenperoxide-solution and the reaction solution is afterwards concentrated to the desired N-Methylmorpholine-N-oxide content. The concentration of the so obtained endproduct can be monitored through measurement of the refractive index or density, in which the endproduct is slightly yellow coloured and as unpurities, nearly undetectable amounts of peroxide, Methylmorpholine and carboxylic groups could be present.

The objective on which the present invention is based is of creating a process for the manufacture of cellulosic mouldings according to the aminoxide process, in which the composition of the spinning solution can be precisely monitored. The regulation of the composition of the spinning solution should be more precisely than it is possible to do this on the basis of a range of refractive indices. In addition to this, a process is to be created for the manufacture of cellulosic mouldings according to the aminoxide process in which the composition of the spinning solution can be regulated with the shortest possible delay, so that any fluctuations which arise in the composition can be kept within narrow limits. Especially a process is to be created for the manufacture of cellulosic mouldings according to the aminoxide process, in which the composition of the different solutions occurring during the course of the process could be regulated even when the measurement of the refractive index is useless, because the light absorbtion or -diffraction of the solutions is to strong. Finally, a device is to be created to carry out the process, by means of which the changes in the composition of the solutions of

the aminoxid process, in fact both the spinning /solutions and the cellulose free, aqueous aminoxide solutions, could be kept in narrow limits or be eliminated. Further advantages can be derived from the following description.

This objective is achieved with the process described in the preamble, according to the invention, that a non-optical property of, at least one of the mentioned solutions is measured and the deviation(s) of the measured value from a certain reference-composition is used for regulating the composition(s) of this/these solution(s). Differing from the above mentioned process including the measurement of the refractive index, according to the invention the deviation of the measured value is given in a narrow limit of tolerance. Deviations of the measured value to the reference value directly set off an intervention at the metering elements of the regulating circuit. The process according to the invention accordingly allows for a substantially stricter regulation of the solution composition than is possible, when the regulation is just activated, when the measured value is leaving the tolerance limit. The measurement of a non-optical property of the solutions does not require the transperancy of the solutions for the wavelength used. So also spinning solutions, which contain defined proportions of additives, such as titandioxide, colour or filling material and aminoxide solutions obtained in course of the process, which are deep coloured because of the impurities, could be controlled and regulated in concern to their concentration.

According to the preferred embodiment of the process according to the invention, the non-optical property of the solution is selected from among the dielectricity constant, the (electric) conductivity, the microwave intensity and - speed, the density, the water content, and the ultrasonic speed. In Process transparent and non-transparent solutions could be controlled by the measurement of the same property,

so the expenditure of measurement and regulation is minimized. The water content can be determined according to the Karl Fischer method.

According to the preferred embodiment of the process the temperature of the cellulose solution is measured at or shortly before or after the measurement of one of the mentioned properties of the solution and the measured value is compensated on the basis of the measured temperature.

Because the temperature of the spinning solution may variate, the measured values must be converted to the reference temperature at which the property values or behaviour values of the spinning solution of the reference composition are known, with which the measured values are to be compared. This reference temperature, to which the measured values are converted, is, for example, 50 °C or 60 °C.

For preference the property of the solution is measured in-line. The in-line measurements allow not only for a rapid determination of the composition of the solution, but also reduce the safety risk engendered by the propensity to decomposition of the solution, due to the possible onset of runaway reactions in hollow spaces of the line (for example in the pipe for taking samples).

In one embodiment of the process according to the invention, recourse is made to the regulation of the composition of the solution in the metering of the components in stage (a), (b) respectively(d). If, for example, a change in the composition of the spinning solution is determined by measurement of the properties, intervention in the metering system of the components cellulose and/ or solvent NMNO/H<sub>2</sub>O is necessary. For the correction of the composition of the precipitation bath solution, it could be necessary to make a recourse of the metering system of the introduced washing

solution or to change the dosage of water to the washing solution.

In another embodiment, recourse is made to the operating conditions of the stage (b) or/and stage (e) regulating the composition of the solution. If the results disclose a shift in the proportion of NMNO/H<sub>2</sub>O, this can be corrected in the stage (b), by the water evaporation being throttled or increased in this stage. The concentration of the aqueous aminoxide solution to be led back in stage (a), can also be regulated by a recourse made in stage of concentration, respectively evaporation.

The objective is further resolved with the device according to the invention, referred to in the preamble, in that in line or in a container, containing the solution to be controlled, a measuring device for the measurement of a non-optical property of the solution is arranged, and that the measuring device together, in line with metering elements or the evaporation device forms a regulating circuit for regulation of the composition of the solution. The measuring device delivers a measured value of the solution, for example the dielectricity constant or the density, which is transformed into signals for the manipulation elements, for example the metering speed of metering elements or the change in the heat performance of the evaporation apparatus.

For preference, the measuring device comprises a device for measuring the temperature of the solution and for temperature compensation of the measured values of the measuring devices. The measuring device accordingly delivers measured values which are already temperature-compensated so that a direct comparison is possible with the property values of the spinning solution of the reference composition, related to a specific temperature.

The invention is described in greater detail hereinafter on the basis of the drawing. The single Figure shows the schematic flow chart of an embodiment of the device according to the invention.

Cellulose pulp containing water and a NMNO/H<sub>2</sub>O mixture are introduced via metering system 1 into a continuous pulping device 2. The device 2 may be a multi-shaft device, such as has been described in DE-C 198 37 210.8. Connected downstream of the device 2 is a dissolving device 3, in which the suspension formed in 2 is transformed into a solution by the addition of heat and under-pressure, under water evaporation. A suitable process for this is known from DE-A 44 41 468.8. Connected to the dissolving device 3 via a line 8 is an extrusion mould 4, through which the spinning solution is extruded via an air gap into a precipitation bath 5. Depending on the type of the extrusion mould and the operations which follow, films, fibres, filaments, or other moulding can be obtained. The moulding 6 acquired in this way is conducted via a withdrawal element 7 for a washing stage 10, in which remaining aminoxide is washed out of the moulding 6.

According to the invention, a measuring device 9 is arranged in the line 8 between the dissolving device 3 and the extrusion mould 4 for the measurement of a non-optical property, for example dielectricity constant or the density of the solution. The measuring device delivers a temperature-compensated signal to a microprocessor 12, which detects deviations from the reference solution and gives manipulation signals via signal line 13 to the metering element 1<sup>a</sup> for cellulose and via signal line 14 to the dissolving and evaporation device 2.

It is obvious from the drawing , that the washing solution coming from the washing stage 10 via line 24 is introduced into the precipitation bath container 5. A measuring device 11 in this line is detecting the composition of the used washing solution in line 24 and regulates via signal line 18 with the microprocessor 16 the valve 17 for the addition of water to washing stage 10. The used precipitation solution is introduced via line 19, which is also containing a cleaning device (not shown), into a evaporation stage 20, in which the the solution is evaporated to a given aminoxide-concentration. The concentration of the evaporated solution is, via measurement of a property by a measuring device 21 and comparison of measured with the reference value via processor 22, transformed into a manipulating signal, which is used for manipulation of the evaporation conditions in evaporation stage 20 via signal line 23.

New Claims

1. Process for the regulation of the composition of solution(s) for the manufacture of cellulosic mouldings, in which

(a) cellulose pulp an a water containing aminoxide are mixed under formation of a suspension,

(b) a cellulose solution is formed from the suspension under the evaporation of water,

(c) the cellulose solution is extruded through an air gap into an precipitation bath, which contains an aqueous aminoxide solution, and there coagulates to form mouldings,

(d) the form mouldings are conducted through an aqueous washing solution in which remaining aminoxide is washed out from the mouldings,

(e) leads aqueous aminoxide solution from stage (c) and/or (d) after concentration back to stage (a), in this

a property of the solutions is measured and according to the measured value the composition of the solutions is regulated,

characterized in that,

at least one property or physical behaviour of at least one of the said solutions is measured and the deviation(s) from the measured value respectively values from a given reference value is used for the regulation of the composition(s) of these solutions, in which the property of the solution is chosen from the dielectricity constant, the inductive conductivity, microwave absorbtion, the density, the water content and the ultrasonic speed.

2. Process according to claim 1, characterized in that, the temperature of the solution is measured at or shortly before or after the measurement of the property and the measured value ist compensated on the basis of the measured temperature.

3. Process according to claims 1 to 2, characterized in that, the property is measured in-line.

4. Process according to claims 1 to 3, characterized in that, recourse is made to the regulation of the composition of the solution in the metering of the components in stage (a), (c) or (d).

5. Process according to claims 1 to 4, characterized in that, recourse is made to the operating conditions in stage (b) and /or (e) for the regulation of the composition of the solutions.

6. Device for the performance of the process according to any of the claims 1 to 5, with a mixing apparatus (2) containing metering elements (1<sup>a</sup>, 1<sup>b</sup>), the mixing apparatus connected to a dissolving and evaporation device (3), an extrusion device (4) connected via line 8 to the dissolving and evaporation device (3, with a downstream precipitation bath (5), at least one washing bath, which contains a diluted aqueous aminoxide solution, and devices for the measurement of a property of the solution(s) to control their composition,

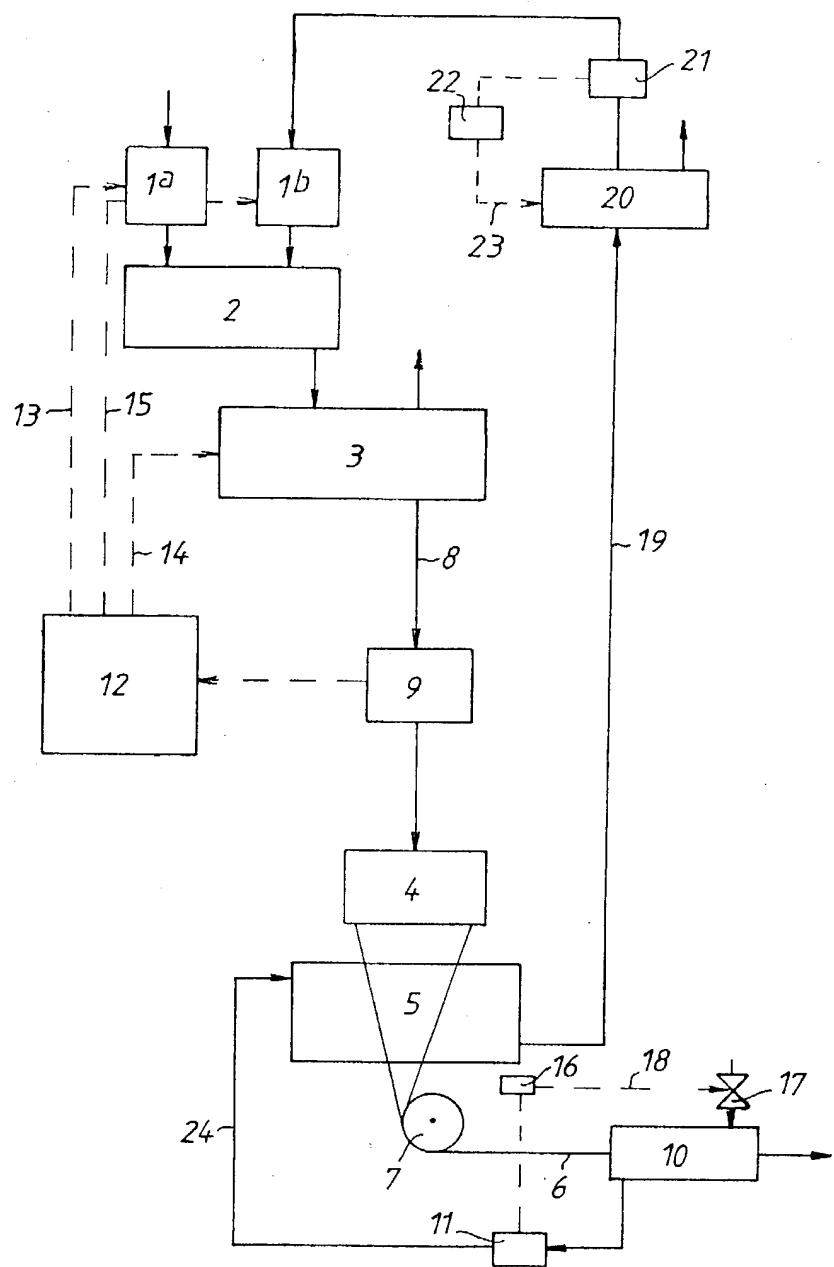
characterized in that, that in the line (8, 19, 24) or in a container, which contains the solution to be controlled, a device (9, 21, 11) for the measurement of a non-optical property of the solution is arranged, and that the measuring device together with metering elements (1a, 1b, 17) or the evaporation devices (3, 20) forms regulating circuits (13, 14, 15, 18, 23) for the regulation of the composition of the solutions.

7. Device according to claim 6, characterized in that, the measuring device (9, 11, 21) comprises a device for measuring the temperature of the solution and for compensating the measured values of the device according to the temperature.

## ABSTRACT

Process for the regulation of the composition of solution(s) for the manufacture of cellulosic mouldings, in which  
(a) cellulose pulp an a water containing aminoxide are mixed under formation of a suspension,  
(b) a cellulose solution is formed from the suspension under the evaporation of water,  
(c) the cellulose solution is extruded through an air gap into an precipitation bath, which contains an aqueous aminoxide solution, and there coagulates to form mouldings,  
(d) the form mouldings are conducted through an aqueous washing solution in which remaining aminoxide is washed out from the mouldings,  
(e) leads aqueous aminoxide solution from stage (c) and/or (d) after concentration back to stage (a), in this a property of the solutions is measured and according to the measured value the composition of the solutions is regulated, characterized in that, that a non-optical property or physical behaviour of at least one of the said solutions is measured and the deviation(s) from the measured value respectively values from a given reference value is used for the regulation of the composition(s) of this solutions. The process allows a precise monitoring of the composition of the extrusion solution.

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## PATENT APPLICATION

DECLARATION AND POWER OF ATTORNEY  
FOR PATENT APPLICATION

ATTORNEY DOCKET NO. 4197-116

As a below named inventor, I hereby declare that:

My residence/post office address and citizenship are as stated below next to my name;

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

METHOD AND DEVICE FOR REGULATING THE COMPOSITION OF SOLUTION(S)

the specification of which is attached hereto unless the following box is checked:

(X )was filed March 26, 1001 as US Application Serial No. 10/089,143 or PCT International Application No. PCT/DE00/03409 filed on September 29, 2000.

I hereby state that I have reviewed and understood the contents of the above-identified specification, including the claims, as amended by any amendment(s) referred to above. I acknowledge the duty to disclose all information which is material to patentability as defined in 37 CFR 1.56.

## Foreign Application(s) and/or Claim of Foreign Priority

I hereby claim foreign priority benefits under Title 35, United States Code Section 119(a-d) or 365(b) of any foreign application(s) for patent or inventor(s) certificate, or 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below any foreign application for patent or inventor(s) certificate having a filing date before that of the application on which priority is claimed:

COUNTRY	APPLICATION NUMBER	DATE FILED	PRIORITY CLAIMED UNDER 35 U.S.C. 119
Germany	199 47 908.9	06 October 1999	YES: <input checked="" type="checkbox"/> NO: <input type="checkbox"/>
Germany	199 49 727.3	15 October 1999	YES: <input checked="" type="checkbox"/> NO: <input type="checkbox"/>
PCT	PCT/DE00/03409	29 September 2000	YES: <input checked="" type="checkbox"/> NO: <input type="checkbox"/>

## U.S. Priority Claim

I hereby claim the benefit under Title 35, United States Code, Section 120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code Section 112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, Section 1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

APPLICATION SERIAL NUMBER	FILING DATE	STATUS(patented/pending/abandoned)

## POWER OF ATTORNEY:

As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) listed below to prosecute this application and transact all business in the Patent and Trademark Office connected therewith.

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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Date

28.05.02

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